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SPECIFICATION FOR READY-TO-USE INSECTICIDES FOR SPOT APPLICATION

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NEW DELHI 110002

Gr 3 *November* 1982

Indian Standard

SPECIFICATION FOR READY-TO - USE INSECTICIDES FOR SPOT APPLICATION

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(Continued on page 2)

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(*Continued on page* 12)

Indian Standard

SPECIFICATION FOR READY-TO - USE INSECTICIDES FOR SPOT APPLICATION

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 26 February 1982, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and Chemical Division Council.

0.2 Ready-to-use insecticides *are* used to control insects in household for spot applications.

0.3 Before proceeding for the quantitative estimation of concentrations as given in 2.2, it would be desirable to ensure that materials conforming to this standard should not include other pesticides.

0.4 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968, and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and tests for ready-to-use insecticides for spot application.

2. REQUIREMENTS

2.1 The material shall be clear and formulated in kerosene-based solvent.

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*Rules for rounding off numerical values (*revised*).

2.2 The material shall be according to any one of the concentrations detailed in Table 1.

TABLE 1 CONCENTRATION OF INSECTICIDES FOR SPOT APPLICATION

SL No.	INSECTICIDE	CONTENT *m/m* *(min)*	METHOD OF TEST, REF TO		
			Appendix	Appendix of IS : 7950-1976*	Appendix of IS : 8496-1977†
(1)	(2)	(3)	(4)	(5)	(6)
i)	Malathion	5·0	A	—	—
ii)	Fenthion	2·0	—	A-2	—
iii)	Propoxur	1·0 2·0	—	—	A
iv)	Lindane	0·5	B	—	—
v)	Chlordane	2·0 3·0	B	—	—

*Specification for fenthion, technical.
†Specification for propoxur, technical.

2.2.1 Malathion, fenthion, propoxur, lindane and chlordane employed in the formulations should conform to IS : 1832-1978*, IS : 7950-1976†, IS : 8496-1977‡, IS : 882-1956§ and IS : 2863-1964‖ respectively.

2.3 **Flash Point (Abel)** — When determined by the method prescribed in IS : 1448 [P : 20]-1960¶, the flash point of the material shall be above 32°C.

3. PACKING AND MARKING

3.1 **Packing** — The packing shall conform to the relevant packing requirements ofindividual insecticide as referred to in IS : 8190 (Part II)-1980**.

3.2 **Marking** — The containers shall bear legibly and indelibly the following information and any other information as is necessary under the Insecticides Act and Rules:

a) Name of the material;
b) Name of the manufacturer;

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*Specification for malathion, technical (*first revision*).
†Specification for fenthion, technical.
‡Specification for propoxur technical.
§Specification for Gamma-BHC (lindane).
‖Specification for chlordane, technical.
¶Methods of test for petroleum and its products: P-20 : Flash point by Abel apparatus.
**Requirements for packing of pesticides: Part II Liquid Pesticides (*first revision*).

c) Date of manufacture;

d) Batch number;

e) Net mass of the contents;

f) Nominal technical content, percent (m/m) ; and

g) A cautionary notice worded as in the Insecticides Act and Rules.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the IS1 Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The IS1 Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by IS1 and operated by the producer. IS1 marked products are also continuously checked by IS1 for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the IS1 Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations'. (***Under Preparation***).

NOTE — Till such time standard under preparation is published, the samples shall be drawn as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in col 4, 5 and 6 of Table 1.

5.2 Quality of **Reagents** — Unless specified otherwise, pure chemicals and distilled water (see IS :1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Specification for water for genera1 laboratory use (*second revision*).

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF MALATHION CONTENT

A-1. COLORIMETRIC METHOD

A-1.0 Outline of the Procedure — The colorimetric method for the determination of malathion involves dissolution of malathion and the formulations in either ethanol or methanol as the preliminary step. In the above formulation, kerosene is used as the solvent. Both rectified spirit and methanol are immiscible with kerosene. Thus the solvent would interfere in the analysis of malathion. Therefore, as the preliminary step, malathion should be extracted with a suitable solvent which would in turn should be preferably soluble in either ethanol or methanol.

A-1.2 Apparatus

A-1.2.1 *Spectrophotometer or Photoelectric Colorimeter* — With a blue filter (420 mm) having 1 cm absorption cell.

A-1.3 Reagents

A-1.3.1 *Malathion* — of known purity.

A-1.3.2 *Acetonitrile* — Boiling range 80 to 82°C.

A-1.3.3 *Ethyl Alcohol* — Anhydrous; or methyl alcohol.

A-1.3.4 *Sodium Hydroxide Solution* — 0·5 N.

A-1.3.5 *Ferric Reagent*

A-1.3.5.1 *Stock solution* — Dissolve 4·0 g of ferric chloride ($FeCl_3 6H_2O$) and 160 ml of concentrated hydrochloric acid in water and dilute to 500 ml.

A-1.3.5.2 *Dilute solution* — Dilute 25 ml of the stock solution with water to 1 000 ml.

A-1.3.5.3 *Copper sulphate solution* (*Copper reagent*) — 1·5 percent (*m/v*). Prepare by dissolving 1·5 g of copper sulphate ($CuSO_4.5H_2O$) in 100 ml of water.

A-1.4 Preparation of Standard and Sample Solution

A-1.4.1 *Preparation of* Standard Solution

A-1.4.1.1 Accurately weigh into a 5-or 10-ml beaker, 0·100 + 0·020 g of pure malathion. Quantitatively transfer it to a 100 ml volumetric flask with ethyl alcohol. Then dilute with ethyl alcohol, to approximately 5 ml below the volume mark. Allow the solution to come to a selected temperature. Record this temperature. Dilute the solution with ethyl alcohol to the mark and mix well. Call this as standard solution A.

> **NOTE** — Standard solution A may be retained for daily use up to two weeks. It is very important, however, that it be adjusted to the temperature recorded above before aliquots are withdrawn.

A-1.4.1.2 Transfer 15 ml aliquot of standard solution A to a 250 ml volumetric flask. Add 2·5 ml acetonitrile and dilute ethyl alcohol to approximately 5 ml below the volume mark. Retain the solution until sample solution (see A-1.4.2) is prepared and is ready for final dilution. At that time, dilute with ethyl alcohol to the mark and mix well. Call this as standard solution.

A-1.4.2 ***Preparation of Sample Solutions***

A-1.4.2.1 Extraction with acetonitrile — Weigh accurately 5 g of the sample and dilute it to 25 ml with hexane and then extract with 25 ml of acetonitrile in a separating funnel. Allow the layers to separate and run acetonitrile layer into a 250 ml volumetric flask. Extract the remaining material in the separating funnel twice using 25 ml of acetonitrile each time. Transfer acetonitrile extract into the volumetric flask. Dilute the contents of the volumetric flask with ethanol up to the mark. Mix well. Transfer a suitable aliquot (which gives a malathion concentration of sample very near to the standard) of the diluted solution into a 250 ml volumetric flask and dilute the same to the mark with methanol. Treat this as sample for analysis.

> **NOTE** — If more then one sample solution is being prepared for assay, then all sample solutions and the standard solution should be diluted to approximately 5 ml below the volume mark as prepared. All should be diluted to the volume mark at the same time.

A-1.5 Procedure

A-1.5.0 To obtain accurate results, it is important that the analysis of standard and sample solutions be carried out simultaneously through the following procedure without interruption.

A-1.5.1 Transfer 25 ml aliquot of standard for analysis and one sample for analysis into separate 250 ml separating funnels. Add 2 ml of sodium hydroxide solution and mix well by swirling gently (do not shake) for 5 to 10 seconds. Allow the solution to stand for exactly 2 minutes.

A-1.5.2 Immediately add 75 ml of ferric reagent and mix well by swirling for 10 seconds. Allow the solution to stand for exactly 5 minutes. During this 5 minutes period, fill two 50 ml graduated flasks to the mark with carbon tetrachloride and after a lapse of 5 minutes, simultaneously transfer the carbon tetrachloride from each volumetric flask to the respective separating funnels. Allow the flasks to drain for one minute.

NOTE-From this point onward, sample for analysis and standard for analysis should be handled separately. Carry each one through the next step without interruption.

A-1.5.3 Add 2 ml of copper reagent (see A-1.3.5.3). Start shaking the separating funnel immediately after addition of copper reagent and shake it for exactly one minute. The malathion-copper complex is unstable in the aqueous phase. The copper reagent should be added with fast delivery pipette and the shaking should be started without loss of any time. Allow the phases to separate. As soon as separation occurs, discard the aqueous phase. Allow some solvent phase to rinse the stem of the separating funnel, then run the clear solvent phase into a small beaker for transferring to an absorption cell or run the clear solvent phase directly into the cell.

A-1.5.4 Measure the absorbance of the yellow solution within 5 minutes at 420 mm using carbon tetrachloride as the blank.

NOTE — Absorbance measurements of both standard and sample solutions should be made at the same fixed time after the end of the shaking period. The yellow colour of the copper-complex in carbon tetrachloride is unstable and hence the colour should be read within 5 minutes after extraction. During this time the absorbance reading drops very slowly.

A-l.6 Calculation

A-1.6.1 The absorbance measurements of the standard and sample solution should agree within ± 10 percent of each other.

A-1.6.2 Malathion content,

$$\text{percent by mass} = \frac{A}{B} \times \frac{m}{M} \times f$$

where

A = absorbance of sample for analysis solution at 420 nm;
m = mass, in g, of pure malathion taken for the preparation of standard for analysis;
f = purity of the malathion used for the preparation of the standard solution;
B = absorbance of standard for analysis solution at 420 nm;
M = mass, in g, of the material taken for the preparation of the sample for analysis.

APPENDIX B

[*Table* 1, *Item* (iv) and (v)]

DETERMINATION OF LINDANE AND CHLORDANE

B-l. REAGENTS

El.1 Isopropyl Alcohol — two concentrations, one 99 percent and the other 50 percent by volume.

El.2 **Metallic Sodium** — Pure, in the form of ribbon or cut in small pieces.

El.3 **Phenolphthalein Indicator Solution** — One percent (*m/v*) in rectified spirit.

B-l.4 Dilute **Nitric Acid** — **50** percent by volume.

B-l.5 Standard Silver Nitrate Solution — approximately 0·1 N.

B-l.6 Ferric Ammonium Sulphate **Solution** — Saturated, aqueous, freshly prepared.

B-l.7 **Standard Potassium Thiocyanate (KCNS) Solution** — approximately 0·1 N.

El.8 Concentrated Nitric **Acid** — sp gr 1·42.

B-2. PROCEDURE

B-2.1 Determination of Total Chlorine Content

B-2.1.1 Weigh accurately a quantity of the material containing about 0·15 g of the lindane or 0·1 gm chlordane in a tared 250 ml Erlenmeyer flask. Add 25 ml of isopropyl alcohol and shake the flask. Introduce 2·5 g of metallic sodium into the Erlenmeyer flask, connect the flask to a reflux condenser and reflux for at least two hours. Shake the flask frequently during the refluxing period. Dissolve the excess metallic sodium by cautiously adding 10 ml of 50 percent isopropyl alcohol through the condenser at the rate of 1 to 2 drops per second. Boil for an additional 10 minutes and then add 60 ml of water.

B-2.1.2 Cool, add 2-3 drops of phenolphthalein indicator. Neutralize by adding dilute nitric acid dropwise and then add 10 ml in excess. Cool the flask to room temperature and quantitatively transfer the contents of the flask to a 250-ml separating funnel. Transfer the aqueous layer to a 500-ml volumetric flask. Wash the contents of the separating funnel thrice using 20 ml water each time. Transfer the washings to the volumetric flask to the mark with water. Mix well.

B-2.1.3 Pipette a 50 ml afiquot of the diluted solution in 250 ml conical flask. Add, with a pipette, 25 ml of standard silver nitrate solution. Coagulate the precipitated silver chloride on a water-bath for 30 minutes, cool the flask and, if necessary, filter the contents of the flask through a fast qualitative filter paper collecting the filtrate quantitatively in a conical flask. Add 5 ml of ferric ammonium sulphate solution either to the cooled unfiltered mixture or to the filtrate as the case may be, and titrate the excess of silver nitrate with standard potassium thiocyanate solution (the end-point is the appearance of red ferric thiocyanate colour).

B-2.1.4 Carry out a blank determination on the reagents using the above method.

B-2.2 Determination of Inorganic Chlorine

B-2.2.1 ***Qualitative Test for Chlorides*** — Shake about 25 ml of the material with 10 ml of water in a separating funnel. Separate the layers and to the aqueous layer add a few drops of concentrated nitric acid and 1 ml of approximately 2 percent (*m/v*) silver nitrate solution. If a white turbidity or opalescence appears, indicating the presence of inorganic chlorine, quantitatively determine the inorganic chlorine in the material.

B-2.2.2 ***Quantitative Determination of Inorganic Chlorine*** — Weigh accurately, by difference, about 10 g of the material in a separating funnel. Extract twice with water using 15 ml each time. Transfer the ‘aqueous extracts to a 250 ml conical flask. Add 5 ml of concentrated nitric acid to the combined aqueous extracts and add 25 ml of standard silver nitrate solution. Coagulate the precipitate as before and titrate the excess silver nitrate with standard potassium thiocynate solution (see B-l.7).

B-2.2.3 Carry out a blank determination on the reagents as before.

B-3. CALCULATION

B-3.1 Inorganic chlorine content, percent by mass

$$= C = \frac{35\cdot46 \times (B - A) \times N}{M}$$

where

B = volume, in ml, of standard KCNS (potassium thiocyanate) solution required for the blank;

A = volume in ml, of standard KCNS solution required for the sample;

N = normality of standard KCNS solution; and

M = mass, in g, of the sample taken for the test.

B-3.2 Organic Chlorine content of the material, percent by mass

$$= D = \frac{35 \cdot 46 \times (B - A) \times N}{M} - C$$

where

B = volume, in ml, of standard KCNS solution required for the blank during the determination of total chlorine content ;

A = volume in ml, of standard KCNS solution required for the sample during the determination of total chlorine content ;

N = normality of the standard KCNS solution;

M = mass, in g, of the sample taken for analysis during the determination of total chlorine content; and

C = inorganic chlorine content, percent by mass (see B-3.1).

B-3.2.1 Lindane content of the material, percent by mass
$1 \cdot 368$ x total organic=chlorine content (*D*) (*see* **B-3.2**)

B-3.2.2 Chlordane content of the material, percent by mass
= $1 \cdot 44$ x total organic chlorine content (*D*)

(Continued from page 2)

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AMENDMENT NO. 1 JANUARY 1985

TO

IS:10119-1982 SPECIFICATION FOR READY-TO-USE INSECTICIDES FOR SPOT APPLICATION

(*Page 4, clause* 3.1) - Substitute the following for the existing clause:

'3.1 Packing - The material shall be packed as per tire-requirement given in IS:8190(Part 3)-1979".

(Page 4, *foot-note with* '**' *mark)* - Substitute the following for existing foot-note:

'**Requirement for packing of pesticides: Part 3 Household pesticides '.

(Page 5; *clause* 4.2) - Substitute the following for the existing clause with Rote:

'4.1 Representative samples of the material shall be drawn as prescribed in IS:10627-1983†.'

(*Page 5, clause* 5.2, *line 2*) - Substitute 'IS:1070-1977†' for 'IS:1070-1977*'.

(Page 5, *foot-notes with* '*'*and*'†' *marks)* -
Substitute the following for the existing foot-notes:

'*Methods for sampling of pesticidal formulations.

†Specification for water for general laboratory use *(second revision)*.'

(AFCDC 6)

AMENDMENT NO. 2 JULY 1988
TO
IS : 10119 - 1982 SPECIFICATION FOR READY-TO-USE INSECTICIDES FOR SPOT APPLICATION

(*Page* 11, *clause* B-3.2.2) — Substitute the following for the existing clause :

'**B-3.2.2** Chlordane content of the material, percent by mass = 1·56 × total organic chlorine content (*D*).'

(AFCDC 6)

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